Sun McMasters and Raven Reitstetter from Dugway Proving Ground on the Testing and Evaluation of Full-Scale Collective Protection Filter System against CWA using validated Advanced Air Purification Filtration System Test Fixture (AAPTF)

## Filter power!

dvancement T&E capabilities to meet the overarching defence programme missions can be accomplished effectively by cooperative Research and Developmental collaboration efforts with Science and Technology (S&T).

To address the need for a verified and validated test fixture to evaluate the performance of full-scale collective protection systems within the Chemical and Biological Defense test community, US Army West Desert Test Center/Dugway Proving Ground (WDTC/DPG) collaborated with PDTESS and the Naval Surface Warfare Division-Dahlgren to develop the new T&E capability.

The AAPTF is a versatile, modular system that is designed to test full-scale CB air-filtration devices against vapour-phase CWAs, CWA simulants, and TICs under controlled environmental conditions. The AAPTF was constructed within a secondary engineering control, denoted as the Chemical Agent Super Chamber (CASC), to contain toxic vapour challenges while providing the

required environmental conditions. Photographs of the CASC and AAPTF are shown in Figures 1&2, respectively. T&E capability of the full-scale CB protection filter using the AAPTF has been verified and validated with consideration of operational controls and safeguards for testing large-scale filters and handling large quantities of hazardous chemical materials.

## **Test Material and Methods**

AAPTF System Description: The AAPTF was designed to operate fully enclosed within a CASC. The exterior dimensions of the CASC are 7.3 x 3.7 x 3.7 m (length x width x height). The AAPTF consists of several modular components that deliver conditioned challenge vapour to the filter housing through 8-inch dissemination lines. Filter housing, butterfly valves, and vapour mixing devices are incorporated in the ductwork of the AAPTF to ensure that the system can challenge a CB filter with homogeneous chemical vapour on demand. The filter

housing is 0.62 m in diameter with a length of  $0.54~\mathrm{m}$  accommodating CB filters up to 0.5metres. Prior to chemical vapour challenging, each CB filter was subjected to leak testing to ensure proper seating and sealing of the full-scale CB filter in the filter housing. The acceptance criteria for the leak test is a downstream (post-filter) reduction in halide vapour challenge concentration of at least 99.9%. A challenge vapourdissemination system for delivery of CWAs, simulants, or TICs is integrated into the AAPTF. The challenge vapour is diluted with conditioned air at a fixed flow rate and passes through the static mixer providing a homogeneous vapour challenge. Challenge-vapour concentration monitoring ports are located upstream and the breakthrough-vapour concentration monitoring ports are located downstream from the filter housing.

Remote Control/Monitoring Methods: Chemical vapour challenge, environmental control, and monitoring were executed remotely (Figure 3). LabVIEW (National Instrument) software was used to operate all test fixture functions such as temperatures, relative humidity, flow rates, and maintain pressure drop across the filter within the rated vendor specification. Pressure drop measurement values across the full-scale CP filter were corrected in accordance with MIL-PRF-51527A to account for the temperature of the conditioned air stream flowing through the filter. The hardware configuration of each instrument control displayed in LabVIEW is depicted in the Super Chamber system diagram shown in Figure 4.

Chemical Materials & General CWA Operation: Chemical compounds used for testing were Isopropyl Methylphosphonofluoridate (GB, CAS: 107-44-8), Bis-(2-chloroethyl) sulfide (HD, CAS: 505-60-2), Opinacolylmethylphosphonofluoridate (GD, CAS: 96-64-0), hydrogen cyanide (AC, CAS: 74-90-8) and cyanogen chloride (CK, CAS: 506-77-4). T&E procedures for handling CWAs and TICs were developed in accordance with (IAW) the safety and



Figure 1. Chemical Agent Super Chamber, the primary containment for the AAPTF



Figure 2. Advanced Air Purification Filtration System Test Fixture (AAPTF)

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security requirements from (1) DA-PAM 385-10, Army Safety Program; (2) DA-PAM 385-30, Mishap Risk Management; (3) DA-PAM 385-61. Toxic Chemical Agent Safety Standards were followed.

Vapour Referee Systems: The AAPTF is integrated with referee instrumentation for quantitative chemical vapour concentration monitoring. Fourier-Transform Infrared (FTIR) spectrometry was used to monitor challenge concentration in real time. Gas chromatography (GC) or GC coupled with mass spectrometry (MS) instruments were used for monitoring effluent concentration in near real time. (1) FTIR gas analyzers [GASMETT DX-4000, Air Quality Analytical Inc] were operated and calibrated based on the in-house FTIR reference spectra generated at the targeted vapour concentration ranges. In all cases, the interferograms were collected from 600 to 4000 cm-1 with a resolution of 8 cm-1. Linear regression coefficients from the calibration plots were greater than 0.998. For quantification, GB molecular vibration absorption occurring at 841 cm-1 (P-F stretching), 926 cm-1 (CH3 rocking vibration of P-CH3), 1019 cm-1 (P-O-C stretching) and 2995 cm-1 (aliphatic CH stretching) were used. GB calibration concentration range was from 34.7 mg/m3 to 4000 mg/m3. GD molecular vibration absorption occurring at 918 cm-1 (CH3 rock of C-CH3), 1019 cm-1 (P-O-C stretching), 1080 cm-1 (pinacolyl moiety) and 2972 cm-1 (aliphatic CH stretching) were used. GD calibration concentration range was from 15 mg/m3 to 4500 mg/m3. HD molecular vibration absorption occurring at 2972 cm-1 (aliphatic CH stretching) was used for calibration and the concentration range was from 25 mg/m3 to 600 mg/m3. For TICs, C-H stretching at 3342 cm-1 and C-N stretching at 2223 cm-1 were used for monitoring at 999 mg/m3. (2) Preconcentrator coupled GCs [MINICAMS®, O.I. Analytical CMS] were calibrated at concentrations ranging from 0.01 mg/m3 to 1 mg/m3 for GB and GD. Both Flame Photo Detector (FPD) and Flame Ionization Detector (FID) were used in this case. HD detection concentration calibration range was from 0.005 mg/m3 to 0.2 mg/m3 using a FPD detector. (3) GC/MS [GC 6890N/MSD 5975C, Agilent Technologies Inc.] configured with a gas phase sampling valve was used. Nitrogen phosphorus detector was also used for TIC detection at calibration ranging from 0.5 to 11 mg/m3.

Safety Measures: Air monitoring systems are integrated throughout the engineering controlled facility including CASC and

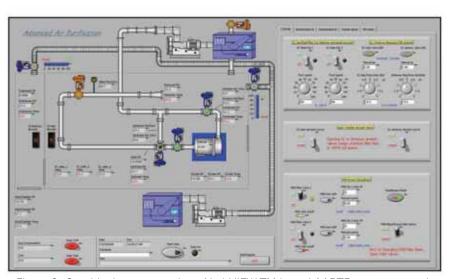


Figure 3. Graphical representation of LabVIEW TM-based AAPTF remote operation

sacrificial filter banks to ensure operational safety of operators (Figure 4). Safety airmonitoring alarm systems are set to warn operators when the concentration of specific vapour reaches exposure actions levels. Military airborne exposure guidelines for unmasked agent workers at the 8 hr timeweighted average in any work shift (Worker Population Limit [WPL]) are 3 x 10-5 mg/m3 for GB and GD. Likewise for HD, it is 4 x 10-4 mg/m3 IAW DA PAM 385-61. MINICAMS® locations for CWA safety air monitoring at the WPL are represented as red boxes in Figure 4. A fixed remote real-time AC detection system, iTRAN (Industrial Scientific Corporation) was set to alarm at 5 ppm with a response time of less than 10 seconds. The CK detection system (Design West Technologies) was set to alarm at 1 ppm with a response time of less than 5 seconds. Locations of these TIC systems are depicted as blue boxes in Figure 2. Emergency response and medical first responder procedures were established for CWA and cyanide antidote. Emergency first aid measures, casualty management procedures, and safety air monitoring plans were also developed to ensure the safety of operators. For AC/CK, the NIOSH CBRN Certification Standard for full face piece air purifying respirators (FR-15-CBRN) was used. M40 masks were used for CWA respiratory protection and additional chemical protective undergarments were worn for HD handling and operation.

## **Results and Discussion**

A new T&E full-scale CB air purification filter capability using the AAPTF has been verified and validated (V&V). During the V&V process attributes and limitations of the AAPTF for T&E were identified. Results from the

representative data plots from the validation trials are shown in Figure 5. We demonstrated that environmental conditions and airflow rates meeting CB filter specifications can be achieved with the AAPTF. Target vapour flow rates from 20 to 500 cubic feet per minute (CFM) were established and maintained within minutes. Airflow rates from the excursion trials verified the consistency and accuracy of the airflow rate. All targeted temperatures were maintained within ±2°C at levels from 0 to 49 oC. Relative humidity (RH) ranging from 6 to 94 % was generated inside the CASC depending on the temperature.

Challenged vapour concentrations ranged from 200 to 2000 mg/m3 and were maintained for a period of up to 45 hours or until the breakthrough was observed. A sparger-type disseminator was used to achieve the targeted vapour challenge concentration for CWAs and liquefied TIC materials were disseminated from cylinders. Purity of all materials used was at least 95%. Effluent concentration level monitored for each vapour type was based on the military exposure guidelines (MEGs) published by the US Army Center for Health Promotion and Preventive Medicine (US Army Public Health Command). Upon completion of V&V, fullscale CB filters were tested against sarin, soman, and mustard as representative CWAs. AC and CK were tested as representative TICs. Furthermore, a material balance approach was taken to validate testing procedures by accounting for the mass difference between liquid chemical material disseminated and consumed. Theoretical values for disseminated vapour concentrations were calculated by multiplying challenge vapour concentrations

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by dissemination vapour flow rate and time duration. The calculated agent mass was compared to the experimental weight loss from the liquid chemical reservoir of the vapour generation device. This mass was also compared to the mass gained by the contaminated CB filter. This mass balance assessment provided appropriate testing characteristics for validating AAPTF operation for large CB filter testing.

Standardized Test and Evaluation
Procedures: Test operating procedures have been standardized for testing full-scale CB filter and V&V trials demonstrated that the procedure is operationally safe and reliable.

(1) Procedures for proper seating of the fullscale CB filter in the test fixture filter housing were developed and the acceptance test criteria were set at a minimum of a 3-log reduction for the halide vapour concentration measured downstream of the filter housing. (2) Material balance was performed by gravimetrically measuring actual mass of chemical consumed in the disseminator reservoir and difference in weights of the test filter between pre- and post-challenge. (3) Full-scale CB filters were conditioned dynamically at least 16 hours at the required environmental conditions prior to chemical vapour challenge. (4) System configuration management and data management

processes were developed and standardized. (5) A standard operating procedure was established for the AAPTF operation and safety procedures and composite risk assessments were developed. (6) Engineering controls of the fixture were verified to be maintainable and sustainable. (7) Redundancy in filtration/blower systems provides a negative air flow through the walk-in hood, referee systems hood, dissemination hoods, and the airlock relative to the surroundings. (8) Safety airmonitoring systems are integrated throughout the chamber and sacrificial filter banks. These fixed alarm systems are set to warn all test participants when the monitored vapour exceeds the worker population limit or permissible exposure limit, respectively. (9) Referee instrumentation to monitor both challenge and effluent concentrations was configured at the required monitoring levels IAW MIL-PRF-51525B. Challenge concentrations were monitored in real time (within 10 seconds) and effluent concentrations were monitored at 4-minute intervals to enable determination of breakthrough time at specified low-level concentrations. (10) Decontamination procedures of the contaminated CP filters for proper disposal and the AAPTF retrograde have been established.

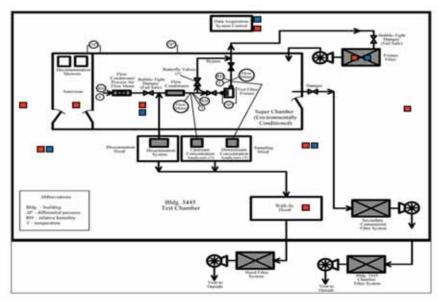
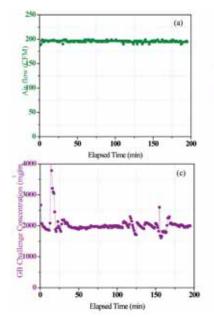


Figure 4. Safety air monitoring locations indicated as red and blue boxes for CWAs and TICs, respectively



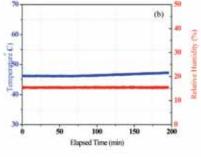


Figure 5. (a) Representative plots of airflow upstream of the AAPTF filter housing targeted at 200 CFM. (b) Relative humidity at 15% and temperature at 46oC. (c) Targeted GB vapour challenge at 2000 mg/m3 refereed concentration.

## Conclusion

The described AAPTF T&E capabilities allow for challenging full-scale CB filtration components with CWAs and TICs. Full-scale CB filter protection performance against CWAs under varying environmental conditions was demonstrated through validation trials and follow-up testing. Through V&V investigation, achievable environmental control parameters for airflow, relative humidity, and temperature were determined; CWA vapour challenge targets that can be reliably maintained have been identified. Testing of expired/used filters against TIC challenges, using the validated AAPTF, is currently being executed. Standardized full-scale CB filter testing using the AAPTF can be extended to perform T&E of other advanced airpurification devices. Assessment of full-scale CB filter performance will provide understanding of the adsorption capacity of activated carbon impregnated with 6% Cu, 6% Zn, 2.5 % Mo, and 0.5% Ag towards varying chemical compounds. Furthermore, the effects of CB filter canister design, vapour residence time, operational conditions, HEPA filter integrations, and vapour flow filtration dynamics on the adsorption performance of the full filter system can be investigated.

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